Functional Guidelines for Evaluating Organic Analysis

CASE No.: 42155   SDG No.: B7FZ0
LABORATORY: DATAC   SITE: RIVERSIDE AVENUE SITE
ANALYSIS: BNA

DATA ASSESSMENT

The current SOP HW-35/SVOA (Revision 1) August 2007, USEPA Region II Data Validation SOP for Statement of Work SOM01.2 for evaluating organic data have been applied.

Data has been reviewed according to TDF specifications, the National Functional Guidelines Report # 3 and CCS Semi-Automated Screening Results Report.

Tentatively Identified Compounds (TICS) for BNA fraction are not validated. They are validated only at the specific request of the data user.

All data are valid and acceptable except those analytes rejected "R"(unusable). Due to the detection of QC problems, some analytes may have the "J" (estimated), "N" (presumptive evidence for the presence of the material), "U" (non-detect) or "JN" (presumptive evidence for the presence of the material at an estimated value) flag. All action is detailed on the attached sheets.

The "R" flag means that the associated value is unusable. In other words, significant data bias is evident and the reported analyte concentration is unreliable.

Reviewer's
Signature: **Shobitha Capil**   Date: **February / 8 / 2012**

Peer Reviewer's
Signature: _____   Date: _____/_____ / 2012

Verified By: _____   Date: _____/_____ / 2012

BNA:

SDG# B7FZ0

1.   HOLDING TIME:

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". The non-detects (sample quantitation limits) will be flagged as estimated, "J", or unusable, "R", if the holding times are grossly exceeded.

The following action was taken in the samples and analytes shown due to excessive holding time.

　　　　No problems found for this qualification.

2.   SURROGATES

All samples are spiked with surrogate compounds prior to sample preparation to evaluate overall laboratory performance and efficiency of the analytical technique. If the measured surrogate concentrations were outside contract specifications, qualifications were applied to the samples and analytes as shown below.

　　　　No problems found for this qualification.

3.   MATRIX SPIKE/SPIKE DUPLICATE, MS/MSD:

The MS/MSD data are generated to determine the long-term precision and accuracy of the analytical method in various matrices. The MS/MSD may be used in conjunction with other QC criteria for additional qualification of data.

　　　　Not applicable.

4.   BLANK CONTAMINATION:

Quality assurance (QA) blanks, i.e., method, trip, field, or rinse blanks are prepared to identify any contamination, which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Trip blanks measure cross-contamination of samples during shipment. Field and rinse blanks measure cross-contamination of samples during field operations. If the concentration of the analyte is less than 5 times the blank contaminant level (10 times for common contaminants), the analytes are qualified as non-detects, "U". The following analytes in the sample shown were qualified with "U" for these reasons:

A)   Method blank contamination:

The following semivolatile samples have common contaminant concentrations reported less than 5x the CRQL. The associated method blank concentration is less than 5x the concentration criteria. Detected compounds are qualified U. Non detected compounds are not qualified. Sample concentrations have been reported at CRQL.

-B7FZ0, B7FZ1, B7FZ2
- **Bis(2-ethylhexyl)phthalate** B7FZ0, B7FZ1, B7FZ2

The following semivolatile samples have analyte concentrations reported less than the CRQL. The associated method blank concentration is less than the concentration criteria. Detected compounds are qualified U. Non detected compounds are not qualified. Reported sample concentrations have been elevated to the CRQL.

-B7FZ1, B7FZ2
- **Phenanthrene** B7FZ1, B7FZ2

B)   Field or rinse blank contamination:
Not applicable.

C)   Trip blank contamination for VOA aqueous samples:
Not applicable.

D)   Storage Blank associated with VOA samples only:
Not applicable.

E)   Tics "R" rejected:
Tentatively Identified Compounds (TICS) for BNA fraction are not validated. They are validated only at the specific request of the data user.

5.   MASS SPECTROMETER TUNING:

Tuning and performance criteria are established to ensure adequate mass resolution, proper identification of compounds and to some degree, sufficient instrument sensitivity. These criteria are not sample specific. Instrument performance is determined using standard materials. Therefore, these criteria should be met in all circumstances. The tuning standard for volatile organics is (BFB) Bromofluorobenzene and for semi-volatiles Decafluorotriphenyl-phosphine (DFTPP).

If the mass calibration is in error, all associated data will be classified as unusable "R".

No problems found for this qualification.

6.   CALIBRATION:

Satisfactory instrument calibration is established to ensure that the instrument is capable of producing acceptable quantitative data. An initial calibration demonstrates that the instrument is

capable of giving acceptable performance at the beginning of an experimental sequence.   The continuing calibration checks document that the instrument is giving satisfactory daily performance.

A)   Response Factor GC/MS:

The response factor measures the instrument's response to specific chemical compounds. The response factor for the Target Compound List (TCL) must be ≥ 0.05, and ≥ 0.01 for the twenty-five analytes with poor response in both the initial and continuing calibrations. A value < 0.05, or < 0.01 for the poor performers indicates a serious detection and quantitation problem (poor sensitivity). Analytes detected in the sample will be qualified as estimated, "J". All non-detects for that compound will be rejected "R".

> No problems found for this qualification.

B)   Percent Relative Standard Deviation (%RSD) and Percent Difference (%D):

Percent RSD is calculated from the initial calibration and is used to indicate the stability of the specific compound response factor over increasing concentration. Percent D compares the response factor of the continuing calibration check to the mean response factor (RRF) from the initial calibration. Percent D is a measure of the instrument's daily performance. Percent RSD must be < 20%, and < 40% for the poor performers. % D must be < 25%, and < 40% for the poor performers. A value outside of these limits indicates potential detection and quantitation errors. For these reasons, all positive results are flagged as estimated, "J" and non-detects are flagged "UJ". If %RSD and %D grossly exceed QC criteria, non-detects data may be qualified "R".

The following analytes in the sample shown were qualified for %RSD and %D:

> The following semivolatile samples are associated with an initial calibration percent relative standard deviation (%RSD) outside criteria. Detected compounds are qualified J. Non detected compounds are not qualified.
> -B7FZ0, B7FZ1, B7FZ2, SBLK66
>
> - **Pentachlorophenol** SSTD020GQ
> -B7FZ0, B7FZ1, B7FZ2, SBLK66

7.   INTERNAL STANDARDS PERFORMANCE GC/MS:

Internal standards (IS) performance criteria ensure that the GC/MS sensitivity and response are stable during every experimental run. The internal standard area count must not vary by more than a factor of 2 (-50% to +100%) from the associated continuing calibration standard. The retention time of the internal standard must not vary more than   30 seconds from the associated continuing calibration standard. If the area count is outside the (-50% to +100%) range of the associated standard, all of the positive results for compounds quantitated using that IS are qualified as estimated, "J", and all non-detects as "UJ", or "R" if there is a severe loss of sensitivity.

If an internal standard retention time varies by more than 30 seconds, the reviewer will use professional judgment to determine either partial or total rejection of the data for that sample fraction.

Not applicable.

8.   COMPOUND IDENTIFICATION:

A)   Semi-Volatile Fractions:

TCL compounds are identified on the GC/MS by using the analyte's relative retention time (RRT) and by comparison to the ion spectra obtained from known standards. For the results to be a positive hit, the sample peak must be within   0.06 RRT units of the standard compound and have an ion spectra which has a ratio of the primary and secondary m/e intensities within 20% of that in the standard compound. For the tentatively identified compounds (TIC) the ion spectra must match accurately. In the cases where there is not an adequate ion spectrum match, the laboratory may have provided false positive identifications.

No problems found for this qualification as per NFG/ CCS reports.

9.   CONTRACT PROBLEMS NON-COMPLIANCE:
Initial calibration:

Initial calibration percent relative standard deviation (%RSD) is outside criteria for the following analytes:

Pentachlorophenol

10.   FIELD DOCUMENTATION: No problems.

11.   OTHER PROBLEMS:

12.   This package contains re-extracted, reanalyzed or dilution runs. Upon reviewing the QA results, the following Form 1(s) are identified NOT to be used.

None.